Consolidation of Ti-SiC Particle-Reinforced Metal-Matrix Composites

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1. Introduction

Discontinuously-reinforced metal-matrix composites (DMMC) have received an increasing amount of attention in recent years due to their improved strength, stiffness and wear characteristics. These benefits make DMMC quite appealing for many structural applications; however, limited ductility and toughness have prevented their widespread use. Limited ductility and toughness are not unique to this class of advanced materials and are shared by such systems as continuous fiber composites and monolithic ceramics. The introduction of weak microstructural features in these materials has long been known to enhance the overall fracture properties. These features have taken on many forms depending on the system involved; for example, microcracking grain boundaries in ceramics (1) and weakened interfacial strengths in continuous fiber composites (2). This approach has proven to be quite effective for a wide variety of materials but remains relatively unexplored for DMMC. It has been assumed that the greatest interfacial strength obtainable will produce an optimized mechanical response. This assumption has not been challenged to any significant degree mainly because of the experimental inability to effectively vary the interfacial characteristics using standard processing procedures.

The ability to control the evolution of matrix-reinforcement interfaces in DMMC through various processing parameters is of critical importance for both scientific investigations and industrial applications. The extremely short time at the compaction temperature that can be achieved with shock consolidation make it a unique method for the minimization (and subsequent systematic variation) of interfacial reactions. Because of the high reactivity of Ti with the readily available reinforcement particles, this system possesses significant potential for gain from improved interfacial control. The fundamental questions concerning the "optimum" interfacial strength for a given DMMC can be systematically investigated with these reactive composite materials.

The objective of this study was to produce fully dense DMMC compacts comprised of a reactive particle-matrix combination with little or no interfacial reactions in the as-consolidated condition. This material can provide unique information regarding the role of interfaces in DMMC.

2. Ti Alloy Composites

Production of continuous fiber Ti based composites has been feasible for many years and reviews of the state of this technology are available, e.g. (3). The major complication in the development of Ti matrix composites, compared to other systems, has been the detrimental interfacial characteristics that are often observed with many reinforcement materials. Most of the investigations that deal explicitly with Ti matrix composites are focussed on the characterization and manipulation of matrix-fiber interfacial reactions, e.g. (3-8). The large reaction zone has effectively precluded the processing of *DMMC based* on Ti alloys.

A primary practical consideration in the analysis of Ti based composites is the operating tempera-

tures to which these materials will be subjected in prospective applications. One potential application is for elevated temperature engine parts where considerable weight savings can be obtained by replacement of superalloy materials with Ti based composites. These parts would have operating temperatures on the order of 500° to 600° C and require good tensile strength properties and creep characteristics after significant exposure at these temperatures. The importance of investigating not only the as-consolidated interface, but also the microstructure of the composite after thermal exposure is clearly recognized. It should be stated that for these expected working temperatures the Ti-SiC interfaces are stable (see Section 3). *The dominant obstacle with respect to detrimental interfacial reactions occurs during the processing of Ti matrix DMMC and is not due to exposure at expected operating temperatures of 600° C.*

3. Ti-SiC Interfaces

The reaction products that have been reported for Ti-SiC interfaces depend on the temperature of exposure. At lower temperatures, below about 850°C, TiC is the only observed reaction product (4), whereas, at higher temperatures Ti_5Si_3 and limited amounts of ternary Ti-Si-C phases are also formed (5-8). Several investigations into the reaction kinetics of Ti and SiC conclude that the reactions are diffusion controlled and suggest several theoretical formulations for the associated reaction zone thickness as a function of time and temperature (5-7). It is important to note that the exposure times associated with these investigations are on the order of hours to hundreds of hours. The processing times encountered in shock consolidation (on the order of microseconds) are not expected to generate significant interfacial reactions.

There would appear to be little doubt that the exposure of the Ti-SiC interfaces to sufficiently elevated temperatures will generate particle-matrix reactions and eventually alter the interfacial strength. At the application temperatures for most Ti alloys, up to 600° C, the kinetics are reduced to such an extent that no reaction products are experimentally observed after exposure times on the order of 1000 hours (4). *Therefore, it is the consolidation temperatures which have limited the development of Ti-SiC DMMC and not the expected service conditions.* These characteristics also make this system ideal for the investigation of the effects of interfacial strength on the far-field mechanical response because the interfacial characteristics can be drastically altered with post-consolidation heat treatments.

4. Mechanics of DMMC

Detailed discussions and reviews on the mechanics of particle-reinforced metal-matrix composites can be found in refs. (9,10). Several investigators have attempted to quantify, using finite-element methods, the stress states at the particle matrix interface, the evolution of interfacial voids and the effects on the far-field response of the generation of these voids (11-13). These studies have been based on experimental evidence limited to the investigation of a composite system with a single characteristic interface. The calculations have, in general, been consistent with the limited experimental evidence; however, comparisons with experimental results for composites with variable interfacial strengths have not been performed.

Extensive interfacial void formation is observed in Al-SiC DMMC only during elevated temperature creep tests. Interfacial voids initiate at the corner of the SiC whisker where the stress concentration is maximum (11-13). The failure is generated by a localization of the strain field and large local tensile hydrostatic stresses. Large compressive hydrostatic stresses along the length of the particle prevents the separation of the interface in regions other than along the far-field loading axis (11). With further straining the particle will then "pullout" from the matrix in a manner similar to that observed in continuous fiber composites. Christman et. al. (12) and Nutt and Duva (13) have published TEM evidence of this mechanism in SiC reinforced 2124 Al composites. Nutt and Needleman (11) have calculated the alterations in the local stress and strain fields associated with the formation of these voids.

It should be clearly stated that this type of failure mechanism is predominant only during creep deformation. Other investigators have reported significant particle cracking and ductile matrix failure during quasi-static testing (9,10). Experimental inabilities in generating a failure mode transition (within a single composite material and loading pattern) has hindered the assesment of each mode on the far-field mechanical response. Failure mode transitions are possible with a reactive particle-matrix composite.

5. Shock Consolidation Procedure and Results

The composites were consolidated with the Keck Laboratory Dynamic Compactor in the Materials Science facility at Caltech. The compactor uses a 3 meter long smooth bore cannon barrel supplied through the courtesy of Aerojet Ordinance. The cannon was designed specifically for the consolidation and recovery of powder materials. A 31.5 mm diameter flyer plate is mounted into a nylon sabot and accelerated by smokeless shotgun and pistol powder (nitrocellulose). For all the experiments discussed in this report, the flyer plate was a 9mm thick disk of 303 stainless steel, having a density of 7.896 gm/cc. Flyer plate velocities are monitored using optical velocimetry techniques. The entire barrel and sample holder are evacuated during the consolidation procedure to a vacuum of $\approx$25 mTorr.

This facility is capable of producing a nearly plane wave shock front with a minimum of reflected stress waves. The degree of shock wave planarity has been investigated previously with experiments on metallic glass consolidation (14). The control of reflected waves has allowed for the consolidation of composite compacts with few cracks and macroscopic defects. This planarity has also allowed the consolidation of multiple cavity green compacts and near-net-shape composite samples (14,15).

Ti-6Al-4V alloy powder was obtained from Powder Metals Inc. and SiC powder was obtained from Electro Abrasives Inc. in 2 powder sizes each. The powders were found to be irregular polygons with average sizes given in Table 1 based on optical examinations at 1000x. The standard deviations for the particle dimensions approached 50% of the given dimension. The Ti and SiC powders were mixed using a medium of petroleum ether at 10 vol.% SiC. The mixture was shaken vigorously, placed in an ultrasonic bath, and shaken again. The ether was then removed by pouring, heating and ventilation. A measured amount of powder was then placed in the target ring and pressed with 10,000 pounds in the shock direction to form a green compact. The green compact was a disk of 32 cm diameter and 1.6 cm in thickness. The distension of the green (theoretical full density/green density) was between 1.9 and 2.0 for all reported cases.

TABLE 1.
Average particle sizes in microns determined by optical microscopy.

Powder	Width	Length	Aspect Ratio
Ti(small)	24	35	1.5
Ti(large)	80	110	1.4
SiC(small)	11	15	1.4
SiC(large)	87	120	1.4

The shock energy deposited in the compact was calculated using standard equations which have been extensively covered in the literature, e.g. (16). For the composite samples, it was assumed that all the deformation in the SiC particles was elastic and therefore, all the shock energy was deposited into the Ti particles. In addition, because of the difficulty in obtaining accurate thermodynamic constants for the particular Ti alloy used, data for elemental Ti was used in the reported calculations.

Exploratory consolidation attempts were then performed to determine the optimum powder size combinations and shock energies. Shock energies were varied from 190 J/gm to 450 J/gm over the

4 possible Ti-SiC powder size combinations. The specific conditions for the consolidation attempts reported here are as follows: flyer plate velocity ≈1050 m/s, shock energy 430 to 450 J/gm. For all the cases investigated, complete densification was observed by optical and SEM investigations. In addition, excellent SiC particle distribution was observed in all compacts. Figure 1 shows an optical micrograph of a typical consolidation.

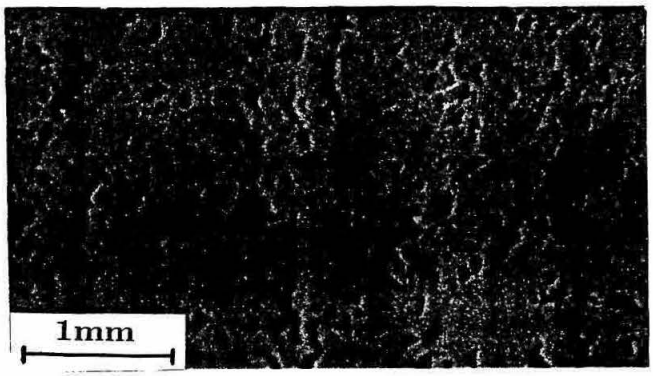

FIG. 1. Optical micrograph of an as-consolidated compact.

The optical and SEM micrographs do not show evidence of interfacial reactions between the Ti matrix and the SiC particles. In addition, preliminary TEM results on composite samples prepared by ion-milling also do not reveal interfacial reaction zones (17). Therefore, one of the primary goals of this study was achieved, i.e. the production of fully dense Ti-SiC composites with minimal interfacial reactions.

The primary defect observed in the compacts was cracked SiC particles as shown in Figure 2(a) for a compact of large Ti and large SiC powders. Particle cracking was minimized for the case of small Ti and small SiC powders, Figure 2(b). Number fractions of cracked versus uncracked particles were determined using optical micrographs and found to range from 0.90 for large Ti and large SiC to 0.05 for small Ti and small SiC. Variations in the shock energy did not appear to affect the number fraction of cracked SiC particles. It should be noted that cracked reinforcement particles are present in most DMMC often due to hot extrusion processing.

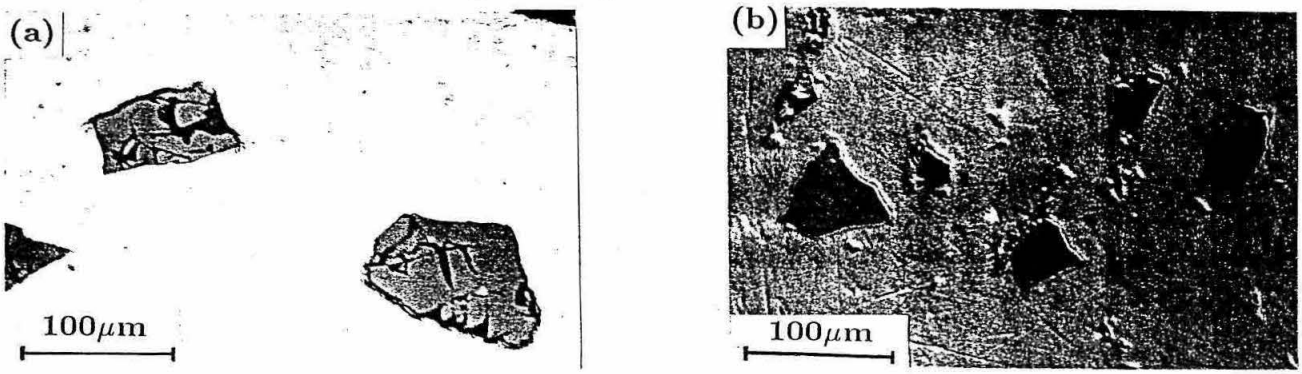

FIG. 2. Micrographs showing the effects of particle size on SiC particle cracking. Figure 2(a) demonstrates the case of large Ti and large SiC and Figure 2(b) demonstrates the case of small Ti and small SiC.

The processing of near-net shape compacts is also possible using this technique. Figure 3 shows a Ti-SiC composite compact with a complex geometry that was consolidated with zirconia powder surrounding the green compact. Zirconia was chosen because it has similar density and shock wave velocity to that of the composite and would be unlikely to bond to the compact during the shock consolidation.

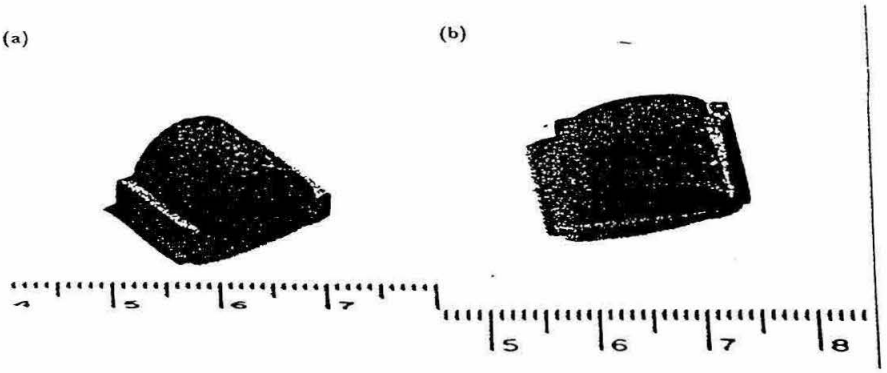

FIG. 3. A near-net shape Ti-SiC composite compact before, 3(a), and after, 3(b), consolidation. Note the retention of the sharp corners and flat surfaces. Note also the lack of compression in directions other than that of the shock wave.

6. Heat Treatment Procedure and Results

A temperature of 850° C is considered standard for annealing Ti-6Al-4V alloys and was chosen as the heat treatment temperature for this reason. As discussed in Section 3, this temperature is sufficient to produce significant reactions between Ti and SiC. Exposure times of 0.25, 1, 4, and 10 hours were chosen to produce a wide range of interfacial thicknesses. The 0.25 and 1 hour heat treatments were applied in a standard atmosphere furnace and the 4 and 10 hour treatments were applied in an argon atmosphere to prevent significant surface oxidation.

Annealing did not generate interfacial reactions of sufficient extent to be observed by either optical or SEM techniques for the 0.25 and 1 hour heat treatments. Exposures of 4 and 10 hours generated detectable interfacial reaction zones and the results are plotted in Figure 4. Also shown are the predicted reaction zone thicknesses from several investigations. The reactions generated in the present study fall within the range of those predicted from the literature.

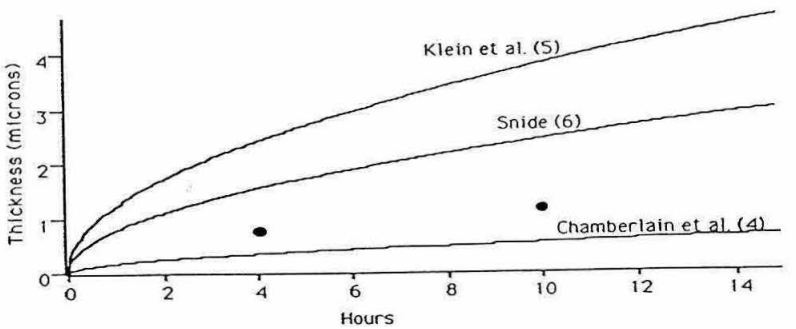

FIG. 4. Calculations of predicted reaction zone thickness from several sources in the literature for exposure times at 850° C and the observed thickness from the present investigation (shown by the closed circles).

7. Summary

This report details the results of our successful attempt to consolidate a highly reactive particle-matrix composite system. This material will be quite useful for the analysis of the effects of interfacial

reactions on the far-field mechanical response of DMMC. The mechanical characterization of this material is presently underway and will be reported in future publications.

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